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(71) Applicant *(for all designated States except US)*: **UNIVERSITY OF BRISTOL** [GB/GB]; Senate House, Tyndall Avenue, Bristol, Avon BS8 1TH (GB).

(72) Inventor; and

(75) Inventor/Applicant *(for US only)*: **QUARINI, Giuseppe, Leonardo** [GB/GB]; Department of Mechanical Engineering, Queen's Building, University Walk, Bristol, Avon BS8 1TR (GB).

(74) Agents: **STONER, G., Patrick** et al.; Mewburn Ellis, York House, 23 Kingsway, London, Greater London WC2B 6HP (GB).

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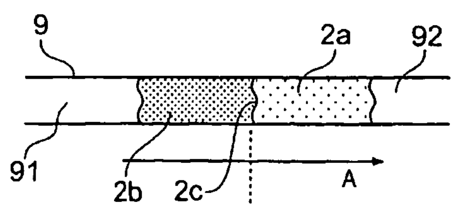
(54) Title: METHODS OF CLEANING, CLEARING AND SEPARATION IN CONDUITS




(57) Abstract: The application describes a variety of special pigging methods and materials, using mixtures of crushed frozen material with its melt liquid, typically also containing a freezing point depressant. One proposal is to form the pig as two adjacent bodies (2a,b) at different temperatures, so that further freezing of the less cold body (2b) by the colder body (2a) raises its solids fraction adjacent the interface (2c) and enables higher-shear cleaning on the conduit wall (9). Other proposals include the use of air voids between ice pigs to enhance their cleaning effect, and the targeting of pigs containing chemically active agents at predetermined locations in a conduit system.

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# METHODS OF CLEANING, CLEARING AND SEPARATION IN CONDUITS

## FIELD OF THE INVENTION

This invention has to do with operations carried out inside fluid flow conduits, in particular pipes and tubes. We envisage application of these new techniques in the flow conduits of processing machinery for food products (including beverages) and food components. Other significant industrial and technical areas include hydrocarbon recovery and the manufacture of both fine chemicals and bulk chemicals and compositions containing them. One aspect of the proposals is concerned with the cleaning or clearing of the conduit interior. Another aspect is a provision of a barrier at the end of a body of fluid passing in or held in the conduit, e.g. to prevent mixing with another adjacent body of fluid in the same conduit. Typically the proposals relate to conduits for liquids.

## BACKGROUND

Our earlier publication WO 01/51224 describes methods in which clearing, cleaning or separating are carried out in a fluid flow conduit by causing a coherent, flowable agglomerate material which consists essentially of particles mixed with a wetting liquid to pass along inside the conduit, spanning its interior so as to contact its interior surfaces. The coherence of the mass, promoted by the wetting liquid, enables the conduit to be blocked or plugged and gives plastic strength to the mass enabling cleaning of the wall. Conversely, the independence of the particles resulting from the mass being essentially non-gelled allows it to negotiate substantial changes in cross-sectional shape as well as sharp bends or even branches of the conduit.

In preferred embodiments, the particles are hard and/or frozen particles and the wetting liquid is or comprises a melt derived from those particles. Ice and water are a preferred combination, with optional additives including a freezing point depressant such as sugar or salt to control the phase equilibrium.

The present proposals develop the use of the methods of WO 01/51224, and in what follows the terms "agglomerate material" and "method of the kind described" are used to refer to the agglomerate material described above and its use, with the above available options and preferences together with any other features as disclosed in WO 01/51224.

SUMMARY OF THE INVENTION

(1) Stiffening

A first aspect addressed here is that of achieving a high stiffness or shear-resistance in the agglomerate mass or "pig".

The ice mass pumped through the conduit in our earlier application is not particularly limited as to its proportion of solid to liquid, or ice:void fraction. In fact a typical crushed ice mass useful for pigging contains of the order of 40 to 50% ice by volume. Pigs with lower solid fractions are very easily pumped, but their cleaning effect - if that is what is wanted - is low. When the ice fraction (here we are using the term "ice" generically, in the sense of any frozen solid whose melt is also present in the agglomerate) rises above about 40 vol%, the agglomerate requires higher pressures to pump and at 50 vol% or above it can be difficult even to get the agglomerate mass into the conduit in the first place. Note that the fraction % figures are generally typical but will depend to some extent on the

compositions and processing conditions. Therefore they are not number-value critical as regards their technical effect; indeed, vol% values herein may equally be taken as wt%.

However a highly shear-resisting stiff pig has the advantage that it generates a powerful cleaning effect at the conduit wall. It would be desirable to be able to use high ice fraction masses.

It is already observed that when a pig of crushed ice is pumped along a conduit, it has a natural tendency to retain its stiffness. Essentially it appears that of the liquid fraction in the mass, a proportion tends to pass to the front of the mass under pumping pressure from behind, because of its low viscosity compared with the shear-resisting interfitting mass of particles. Thus the ice fraction may be maintained, and may increase towards the tail end of the pumped mass.

The ice fraction naturally depends on the temperature and basis material of the mix, also on the concentration of any freezing point depressant present. The lower the temperature, the higher the ice fraction for a given concentration of freezing point depressant. The higher that concentration, the lower the ice fraction at a given temperature.

A first aspect of our proposals here is to establish in the conduit and to pass along it an agglomerate mass having axially-adjacent regions of different temperatures. Preferably the agglomerate material for the respective regions is prepared separately to achieve the different properties. In the conduit the two parts of the mass make a direct interface. Preferably the initial temperature difference between the two parts of the mass is at least 5°C, perhaps 8 or 10°C or more.

However both parts of the mass should have a solid fraction consistent with their being driven along the conduit, preferably by fluid pumping into the conduit behind the plug body. Preferably the solid fractions in the two regions are comparable or the same, say within 5 vol% (or 5 wt%) of one another. This may be achieved by providing a higher level of a freezing point depressant in the colder region material; the other material may contain less or none at all. Or, one portion may have a more potent depressant than the other, or be of a different material altogether.

At the interface between the regions, there is heat transfer, with some melting of the colder material by the less cold, and with some extra solidification leading to increase in solid fraction in the less cold region. For reasons mentioned previously, additional melt can usually pass forward out of the mass e.g. under hydrodynamic forces. This is not true for the solid particles, and a marked rise in solids fraction appears in a localised band of the less cold material adjacent the interface.

As mentioned, increases in solid fraction above a certain level lead to very steep increases in shear resistance. The result is a pig consisting mostly of readily-pumpable agglomerate, but entraining a relatively axially local region of higher-solids, strongly shear-resistant or stiff material. This can give a powerful cleaning effect at the conduit wall while avoiding the practical difficulties that would be encountered were one to attempt pigging with an agglomerate mass having a correspondingly high solids fraction throughout, and from the outset.

Preferably the colder region leads the less cold region as the plug body is driven, although this is not

essential. Indeed, there may be more than two regions of alternating temperature, giving more than one high solids fraction band.

The pig as a whole, and/or the mentioned temperature-distinct regions thereof, may satisfy the suggested relationships between pig length and conduit dimension as described elsewhere herein.

(2) Use of gas

In a second aspect of the new developments, which may be combined with any others herein, we have found that for a given quantity of a given agglomerate material, better cleaning and separation effects can be achieved if the material is used adjacent a void in the conduit occupied by air or other gas. Where the agglomerate material meets a liquid directly, there is a degree of mixing. Separation of the agglomerate material from other materials in the pipe naturally eliminates or reduces such mixing. However we also find that the interface between the material and adjacent gas tends to be sharper than an interface with liquid. Also, in the context of cleaning/clearing, we find that the cleaning effect at the wall of the conduit, e.g. to remove residues or contaminants there, is greater when a phase discontinuity moves along the wall, and that the effect is greater for a phase interface between gas and the agglomerate material than it is at an interface between liquid and the agglomerate material.

Thus, this aspect of the invention provides a method of cleaning, clearing or separating in a fluid flow conduit by causing a body of the agglomerate material to pass along inside the conduit, spanning its interior to contact its interior surface, characterised in that the body of the material is passed along the conduit adjacent

a gas-filled (e.g. air-filled) void, the void being between said body and another such body, or between said body and other flowable material in the conduit. Preferably the direction of passage has the leading face of the agglomerate material body at the moving interface with the adjacent void.

The extra effectiveness of a gas/agglomerate interface in cleaning and/or separation can be exploited further to increase the effect achieved with a given amount of agglomerate material.

In particular, in a preferred embodiment agglomerate material is passed along the conduit as two or more portions separated by void, and with one or more of the portions presenting a leading face which faces onto a void, at least where it contacts the conduit's interior surface and preferably right across the conduit so that the portions are completely separate plugging bodies.

In one embodiment an agglomerate body follows a body of process liquid in a conduit with a void in between the two. Additionally or alternatively, two, three or more bodies of agglomerate material proceed as a set along a conduit, separated from one another by voids. As in previous proposals, such a set of agglomerate bodies may pass along a conduit as a barrier or clearing/cleaning body at one end of a body of other flowable material in the conduit (process liquid), or between two bodies of other flowable material (process liquids) in the conduit, or as a means of cleaning the interior of an otherwise empty conduit.

The bodies of agglomerate material (or portions as mentioned above) passed along the conduit may be at least one, two, three or four pipe diameters in length. Conversely, they are preferably not more than twenty pipe

diameters long, preferably not more than ten pipe diameters long and commonly not more than five pipe diameters long.

Void length, between a body of agglomerate material and an adjacent such body or adjacent body of other fluid in the conduit, is preferably at least 0.5, 1 or 2 pipe diameters. Conversely, the void length is preferred not to be above ten diameters, more preferably not more than five diameters. For non-circular conduits, the reference to diameter can be taken as $2\sqrt{(A/\pi)}$ where A is the cross-sectional area of the conduit. The skilled person will appreciate that for a given purpose in the conduit, the number, size and spacing of plugs of agglomerate material can be selected in accordance with routine tests to optimise the desired effect.

Independently of the above (although it may be used in combination), we propose another development relating to the use of gas in methods of the kind described. In this aspect, blended gas is included in the agglomerate of the particles and wetting liquid. For example, air is blended in a (water) ice agglomerate by stirring or other agitation. These gas-blending techniques are known as such; they are used for example in the production of flavoured ice "slush" confectionery products. One very effective technique is to disperse gas (e.g. air) into the agglomerate material (e.g. water/ice/freezing point depressant) by injecting the gas in it, (e.g. from one or more nozzle, spray or jet openings) air bubbles, under pressure. Substantial volumes of air can be entrained in the agglomerate, and advantages are available. The gasified agglomerate plug tends to be both stiffer and more compressible than a gas-free plug. Also, since a given volume of agglomerate contains a smaller weight of

water, less material is used. This may be significant for e.g. the disposal of contaminated material, where the volume and hence any effluent problem (e.g. biological oxygen demand) is correspondingly less.

(3) Scouring additives

A further proposal, usable in combination with any other proposal herein or independently, is to use an agglomerate mass of the kind described containing a particulate non-melting scouring material such as a sand or grit. Embedded particles of sharp, hard inorganic material augment the cleaning effect at the conduit wall.

(4) Residence time/scrubbing

A related proposal, again usable either independently or in conjunction with any of the other proposals, is to augment the cleaning effect at a given location in the conduit by repeated passes of an agglomerate mass at that location. In particular, passes repeated by a reciprocating movement of the agglomerate mass at the chosen position which is local relative to the total travel of the mass in the conduit. This reciprocation can be driven by appropriate driving of a pump or pumps, and enables particularly fouled regions to be dealt with.

The above may also be an example of a further or more general proposal, which is to increase the contact or residence time of an agglomerate mass at a predetermined position in the conduit relative to elsewhere in the conduit. This is particularly of interest where the agglomerate mass consists of or contains a chemically active material whose action is particularly desired at the location in question, and/or particularly undesired elsewhere, and/or transient in effect. The procedure may involve pumping to drive the

agglomerate mass to the predetermined location and then slowing (preferably stopping) pumping for the active agent to exert its effect locally. This may however be combined with local reciprocation as mentioned above.

The selected location might be any part of a duct or conduit with a special cleaning or treatment requirement. It might be for example a "difficult" topology such as a dead zone not normally subject to flow e.g. a dead leg or side arm. By holding the active agglomerate pig adjacent this region, there is time for diffusion of the active agent to take place and treat the region. Typically this is accompanied by some melting of the agglomerate which nevertheless maintains its integrity and can be pumped away as a body so that much or most of the active ingredient is promptly removable from the system.

Such active ingredient might be an inorganic or organic sterilising medium, bleach or biocide. While normally it is preferably soluble in or constitutes part of the freeze/melt system of the agglomerate, this is not strictly necessary and these agglomerates can carry immiscible substances.

The above two proposals involve controlling the action of the "pig" at a predetermined location. Since ducts and pipes are normally opaque it is then necessary to have a means for determining the position of the pig. In many cases simple determinations of pumping rate and "time of flight" are sufficient to establish the pig's position with good accuracy. When these data are not easily obtained, or further certainty is required, detection means may be provided at or adjacent the location(s) in question to detect the presence and/or arrival of the pig.

Since an agglomerate mass of the kind described is typically below ambient temperature, the detection means may comprise a temperature detector on the conduit wall, such as a thermocouple. However unless an invasive detector is used, normally not preferred, there will be a significant lag in detection due to time taken for conduction through the pipe wall. Thus, an alternative detection means uses mechanical vibratory transmissions into the conduit, e.g. acoustic or ultrasound, to detect promptly a change in transmission characteristics of the pipe interior due to the presence of a different material inside.

The detection device(s) can be used solely for confirmation, or in a control mode connected to switching circuitry responsive to the detected arrival of the agglomerate mass at the location in question to send an operating signal e.g. to stop pumping, to start a timer for the predetermined residence time or the like.

<u>(5) Special materials</u>

A further aspect herein includes some refinements in the preparation of the substances contained in the coherent, flowable agglomerate material used in methods of the kind described.

In general terms, what we propose in this aspect is that particles of the material, and preferably all or substantially all of the particles, are structured with a gradation or progression of melting point from the inside to the outside of the particles. Thus, where the particles are of a frozen material which is a mixture, and whose melting point varies according to the proportions of the components of the mixture (e.g. the proportion of a freezing point-adjusting substance dissolved in a main liquid), the relative proportions of

those materials vary from the inside to the outside of the particles.

Furthermore, we prefer that the wetting liquid around the particles continues the freezing point/melting point trend in the particle structure. That is, where the particle material decreases in freezing point from the inside to the outside, the surrounding wetting liquid has a still lower freezing point. Or, where the particle material increases in freezing point from the inside to the outside, the wetting liquid has a still higher freezing point. Particular behaviours of these two modes are discussed below.

We prefer the use of aqueous or substantially aqueous materials. The nature of a material mixed with/dissolved in water, and acting as a freezing point adjuster (especially, depressant) is not particularly limited. It can be selected according to convenience and the intended use. Thus, one of the most effective and least expensive freezing point depressants is common salt (NaCl), which reduces the freezing point of brine by about 1°C for every 1% increase in salt concentration. The saturated solution is about 23 wt%. In other contexts, different freezing point adjusters might be useful and these can have selected functional attributes. They may be cleaning agents, for example. Peracetic acid is a compound which acts to sterilise/disinfect, and is environmentally friendly in the sense that its degradation products are carbon dioxide and water.

Preferably, as in earlier proposals, the wetting liquid corresponds to a melt liquid deriving from (or consisting of) a melt of the particle material, or (alternatively stated) a mixture comprising the same

components as the particle material, but in proportions such as to be liquid at the prevailing temperature.

This aspect of the invention includes methods of clearing, cleaning or separation carried out in a fluid flow conduit, of the kind described, in which the flowable agglomerate material has the properties described herein.

The ambient temperature for the process may be at or above the highest freezing point of the particle material, but preferably not more than, say 20 or 30°C above.

A related aspect is a method of preparing such a material, including the preparation of the particles having the graded or progressive freezing point properties in their structure, either as an independent method or as a preliminary step for the clearing/cleaning or separating method. The particle preparation may involve for example plural stages of freezing from freezable liquid containing the mentioned components, the method comprising successive stages of freezing - generally rapidly, to 'trap' the solute - from respective liquid sources prepared at different compositions. These may be discrete stages, so that the particles are layered around a core, e.g. in two or three or more layers (including the core). Or, there may be continuous variation of the liquid source composition. The level of change of composition will depend on the properties required and the materials involved, but may be substantial. For example, in a mixture of a major liquid (e.g. water) with a minor freezing point-adjusting component (e.g. salt, sugar, peracetic acid) the proportion of the minor component in the particles at their surface may be at least 30% different, and perhaps

at least 40% or 50% different, from the concentration of that component half-way to the particle centre.

(6) General

As in WO 01/51224, a preferred material for all purposes is a mass of wet coherent ice (water-based ice) particles which preferably contains a freezing point depressant. Other frozen liquids may be used, in particular when they have melting points in the vicinity of the intended operating temperatures. Also, again as disclosed in the previous application, freezes of an actual process liquid, or of liquid specially selected for compatibility therewith, may be used.

The present proposals do not interfere with the original proposal's utility in conduits which are non-uniform in size or shape. Thus, the above processes in general may be carried out along conduits having one or more of

- substantial changes in cross-sectional area, e.g. greater than 20%;
- internal obstructions, such as probes or sensors, projecting into the conduit interior;
- branching or merging of conduits.

In the case of branching, the procedure may involve the fluid agglomerate mass dividing to follow different branches simultaneously, and intervening voids as prescribed herein may divide similarly.

Conduit sizes are not particularly limited, but in typical processing machinery, e.g. food processing machinery, the conduit size (diameter, or other transverse dimension as suggested above) ranges from 10mm to about 100mm.

The process may be used in a variety of contexts, for example food processing plant, hydrocarbon recovery

in the oil industry, petrochemical processing, bulk chemicals, and in the fine chemicals industry, e.g. dyes and pharmaceuticals, also the nuclear industry.

DESCRIPTION OF EMBODIMENTS

The application of our proposals above is now illustrated further with reference to the accompanying drawings, in which

Fig. 1 shows schematically a two-temperature crushed ice plug in a conduit;

Figs. 2, 3 and 4 are respectively profiles of temperature, salt concentration and ice volume fraction along the Fig. 1 crushed ice plug;

Fig. 5 shows pigging using an air void to clear a conduit;

Fig. 6 shows pigging using two plugs and two air voids, separating two process liquids in a conduit;

Fig. 7 shows an alternative to Fig. 5 using a single void;

Fig. 8 shows the use of blended air in a crushed ice pig;

Fig. 9 shows gradations of freezing point in a system of frozen particles in liquid, and

Fig. 10 shows schematically a piping system with a region having a special cleaning requirement.

(1) Stiffening

A first embodiment is discussed with reference to Figs. 1 to 4. The aim is to achieve a high shear rate, and hence a strong cleaning effect, at the inside wall surface of the conduit 9. The procedure exploits the physical chemistry associated with freezing processes, and the involvement of freezing point depressants such as salt or sugar.

Two different slurries of ice in brine are prepared separately, one with a high salt concentration (e.g. 10 to 20 wt%) in the starting brine and the other with a lower concentration e.g. 0 to 10 wt%, preferably at least 5 wt% lower than the higher concentration brine. Respective ice slurries are prepared to substantially similar solids fractions. The stronger brine requires colder freezing for this. A preferred solids fraction in the slurries is in the range 40 to 60 vol%, typically 45 to 55 vol% for water ice. These levels of solids are about as high as can conveniently be deployed in most piping systems. The saltier slurry might be at about -15°C, the less salty one at -5°C. A front plug 2a and a rear plug 2b are loaded into the conduit 9 adjacent one another, forming an interface 2c.

Figs. 2, 3, 4 show the profiles of temperature, salt concentration and ice fraction along the ice pig 2b,2a. In Fig. 1 the direction of pumping is indicated by the arrow A; this is by means of a conventional pump acting on a process liquid or a propellant liquid 91 in the space behind the plug. The space 92 ahead of the plug may be void for cleaning, or occupied by some process liquid that needs to be cleared from the conduit 9.

Fig. 2 shows how the temperature in the conduit drops from ambient at the location of the ice pig. The front zone is the coldest. At the interface 2c between the front and rear zones 2a,2b there is heat transfer. The portions of the front and rear portions of the pig adjacent the interface 2c are respectively warmed and cooled by one another i.e. their temperatures approximate, as indicated by the gradient in Fig. 2. There is some melting at the front of the interface and freezing behind it. The differences in salt

concentration promote these changes. Any excess melt water from melting in front of the boundary 2c tends to be swept through the pig structure by the pressure behind, towards the front space 92. However immediately behind the junction, the extra freezing raises the solids fraction as shown by the peak in the centre of Fig. 4. Even if this rise in solid fraction is of only a few percent, it occurs in a range at which stiffness increases very rapidly or exponentially with increase in solid fraction. A band of very high stiffness material therefore arises, carried along by the surrounding lower-stiffness plug, and is able to exert a very powerful cleaning effect on the conduit wall.

(2) Use of air voids

With reference to Fig. 5, a liquid food product 1 is present in a conduit 9, e.g. a 25mm diameter (D) processing pipe. To clear the body of process liquid 1 from the pipe 9, a mass of wet crushed ice, incorporating sugar or salt as freezing point depressant, is introduced into the pipe as a plug body 2 with an air-filled void between the ice and the end of the liquid product column 1. A pump is used to drive the flow in the direction of the arrow, whereupon the air-filled void is found to be maintained between the ice 2 and liquid 1 with good definition. The leading face 6 of the ice body 2 is, we find, sharper than the leading face obtained when the ice body directly contacts liquid ahead of it. The passage of the leading edge of the ice body 2 over the interior surface of the pipe 9 is found to have a good cleaning effect, better than that of an ice body contacting directly with the liquid without an intervening void.

Fig. 6 firstly indicates an extension of the above concept, exploiting the findings to a greater degree.

Here the ice agglomerate is divided into two bodies 2, 2' with a first void 3 between the leading ice body 2 and the product liquid in the pipe, and a second void 4 between the two ice bodies. As the liquids are pumped along the pipe 9, the cleaning effect of the leading edges 6 of the ice bodies is repeated, achieving a greater level of cleaning than would be obtained with the same amount of ice combined in a single body.

Fig. 6 also shows the presence of a second process liquid 5 in the pipe behind the second ice body 2', effectively separated from the first liquid 1, and entering a pipe interior which has been effectively cleared of residues of the first liquid 1. Note that the second liquid 5 directly contacts the rear face of the second ice body 2'. An air void could be provided here, but would have less effect.

It will be appreciated that if appropriate the cleaning effect can be increased by passing the comminuted wet ice as three or more bodies separated by voids.

In some cases the behaviour of the process liquid 1 may make the maintenance of a void difficult. In this case, a first ice body 2 may form an interface with the liquid 1 without any void. The first body 2 creates an effective barrier at the end of the liquid 1, and a rear face 7 apt to form a void 4 before a following ice body 2' whose ice/air leading interface 6 will have the enhanced cleaning effect referred to previously. Further bodies of ice may follow after further voids if wished.

(3) Dispersed air

In another experiment, the crushed ice material, prepared from water containing dissolved sodium chloride acting as freezing point depressant, was subjected to

agitation to entrain a substantial percentage of air in the material. A plug 20 of this material - see Fig. 8 - was then introduced into a conduit 9 against a body of process liquid 1 and found to perform just as well as the non-aerated bodies in blocking the pipe and clearing the wall surface. Also, the less dense aerated material was noticeably stiffer (more viscous) than the non-aerated material, and maintained a notably good contact against the pipe's interior surface.

No surfactant was needed to keep the air entrained in the ice preparation, so this benefit was achieved without any potential environmental disadvantage.

It will be appreciated that the aerated ice preparation can be used with the "voids" proposal outlined above, and in any of the other proposals herein.

(4) Controlled gradation of freezing point

In one optional procedure mentioned above, the concentration of a freezing point depressant increases from the inside to the outside of the particles, and the surrounding liquid is still more concentrated. The properties can be described with reference to Fig. 9, which shows particles 105 surrounded by a wetting liquid 104. The particles are shown in schematic cross-section, to show a core 101, intermediate layer 102 and outer layer 103 concentrically superimposed. Particles of this structure might be made for example from aqueous brine, by successive passes through an ice making machine, progressively increasing the NaCl concentration in the brine supply. In an example there are three passes, the NaCl concentrations in the brine in the three stages being respectively 3%, 5% and 8%. The NaCl concentration in the brine which wets the resulting mass of particles is a few % more, say 10%.

Even with flash freezing the NaCl concentrations in the frozen particle layers 101,102,103 do not correspond exactly to the source brine concentrations but are correspondingly graded so that the core 101 has the lowest salt concentration and the highest freezing point, the outer layer 103 has the highest salt concentration and the lowest freezing point, and the intermediate layer 102 has intermediate properties. This mass of wet particles has a very valuable property of stability. There is little tendency for the particles to be lost by melting, and little tendency for the particles to stick together by coalescence. While there is in principle a unique temperature of total stability, a rise in temperature will lead to melting of the outermost layer but the tendency to melt will then decrease as the freezing point rises. Conversely, because the surrounding strong brine is reluctant to freeze, temperature variations below the above-mentioned unique temperature do not tend to lead to coalescence of the particles.

The particulate mass prepared in this way therefore has a long life time in use, without being sensitive to temperature variations, and may indeed be kept for some time before use without losing its valuable flowable and particulate nature.

In a second embodiment, the concentration gradients described above are reversed. The highest concentration of freezing point depressant (e.g. NaCl) is now at the centre core 101 and the lowest in the outermost layer 103. The particle has the highest freezing point at the outside and the lowest in the centre. The surrounding liquid 104 has an even lower concentration of depressant, i.e. higher freezing point. This is an unstable

situation. Irrespective of temperature, there is a tendency for the particles to melt and for the adjacent solution to freeze. There is no need for ambient temperature to vary in a particular direction for this to happen. The speed of the phenomenon is controlled by heat transfer in the solid and liquid.

While this agglomerate clearly lacks certain advantageous properties of the first embodiment, i.e. lifetime and storability, its particular properties may be put to use. Because of the tendency of the fluid surrounding the particles to freeze, to an extent depending on the level of initial cooling when preparing the materials, there is the ability for freezing liquid to bridge between particles and for a whole mass of particles then to "stiffen" i.e. its effective viscosity increases. As mentioned, this is a transient phenomenon whose timing is dictated by heat transfer in the material. When this material is deployed in the clearing, cleaning or separation method to which the invention relates, it is possible to control the time of arrival of the corresponding "ice pig" at a specified region of the apparatus, e.g. part of a processing pipe, where the high viscosity or stiff body is required, e.g. for scrubbing an area particularly liable to fouling. Because of the time dependency of the material's behaviour, it need not however show that high degree of stiffness or viscosity while it is being pumped to the location concerned.

(5) Localised action

Figure 10 shows schematically a conduit 9 including a "problem region" X including components presenting cleaning difficulties. These are exemplified here by a closed-off dead leg of piping 61 and a closed valve T 62.

The skilled person will appreciate that there are many other elements of a flow system that may present special local cleaning difficulties of various kinds.

An ice pig 2, which may be of any of the kinds described herein or in our earlier application, is introduced into the conduit at S and pumped through the conduit 9 (by pumping water in behind it) until it reaches the problem region X. Pumping is then stopped so that the pig 2 remains at region X.

To do this it is necessary to know when the pig 2 lies at region X. This can be done by direct assessment of the volume pumped in behind, or by timing e.g. based on previous trials, or in various other ways. Fig. 10 shows a positive determination using ultrasound transducers 80 disposed immediately downstream of the region X. These can detect promptly, by noting the changed transmission characteristics of the conduit interior, when the leading end of the pig 2 arrives. This ensures, having determined the size of the pig 2 at the outset, that the pig 2 lies in the region X.

For closed legs there is no flow. The pig cannot clean by its normal shearing mode. Nevertheless it may be very important to clean such sites. Conventionally they might be cleaned by filling and flushing the entire system using a cleaning agent, allowing sufficient residence time for even the dead portions to be sterilised. However such extensive use of cleaning agent may be undesirable for a number of reasons. Firstly, there is a general desire to minimise the use of potentially damaging or toxic chemicals, particularly in pipework used for the preparation of consumer products. Secondly, there may be systems in which the cleaning

agent, although needed at one location, would be positively harmful to another part of the system.

By incorporating the cleaning agent in an agglomerate mass of the present kind, its effect can be localised. It can be driven rapidly to the target area, scarcely affecting the regions passed en route. When positioned at the target area, it can be left for long enough to allow some melting and diffusion of the agent, for a length of time sufficient to achieve the desired effect. It nevertheless maintains its integrity, so that after the desired cleaning it can once again be driven briskly out of the system. By far the larger part of the active agent can then be recovered, and in compact form.

Fig. 10 also shows schematically a further local cleaning option, not necessarily combined with the chemical treatment described above. Where a region of the conduit has particularly persistent residues on the wall interior, the pig 2 on reaching that position is moved backwards and forwards (by appropriate control of the pumping system) to scrub the wall and give additional cleaning according to need. This is indicated by the arrow Z in Fig. 10.

Note that apparatus specifically adapted for putting any of the above processes into effect is an aspect of the invention. This may apply to the conduit system itself, e.g. having detector means to detect a pig at a specific cleaning location of a conduit. Or it may apply to auxiliary equipment, especially a freezing machine adapted to prepare separate frozen slurries at two different temperatures for the stiffening described above, preferably at the same time.

CLAIMS:

1. A method of cleaning, clearing or providing a movable barrier in a conduit, comprising passing along inside the conduit a plug body of a coherent flowable agglomerate material consisting essentially of cohering particles of frozen solid material mixed with a liquid which comprises a melt liquid corresponding to the frozen material of the solid particles,

characterised by use of a said plug body which comprises first and second axially-adjacent portions, the agglomerate material of the first portion having a lower temperature than that of the second portion to provide a region of increased solids fraction in the second portion adjacent the interface between the portions.

2. A method according to claim 1 in which the temperature of the first portion is at least 5°C lower than that of the second portion.

3. A method according to claim 1 or 2 in which the solids fractions of the agglomerate materials of the first and second portions are substantially the same.

4. A method according to any one of the preceding claims in which the solid and melt materials of the first and second agglomerate materials are the same except that the first agglomerate material contains a higher level of and/or a more powerful freezing point depressant than the second agglomerate material.

5. A method according to any one of the preceding claims in which the agglomerate material consists

essentially of ice mixed with water, optionally with a freezing point depressant.

6. A method according to any one of the preceding claims in which each of the first and second regions is at least as long as the conduit is wide, and preferably at least twice as long.

7. A method according to any one of the preceding claims in which the solids fractions in the agglomerate materials for each of the first and second portions are at least 40% by volume.

8. A method according to any one of the preceding claims in which the solids fraction in the increased solids fraction region is above 50% by volume.

9. A method of cleaning, treating, clearing or providing a movable barrier in a conduit, comprising passing along inside the conduit a plug body of a coherent flowable agglomerate material consisting essentially of cohering particles of frozen solid material mixed with a liquid which comprises a melt liquid corresponding to the frozen material of the solid particles,

characterised by

driving the plug body to a predetermined axially-localised region of the conduit having a particular treatment or cleaning requirement and maintaining it at that region for longer than at surrounding regions.

10. A method according to claim 9 comprising reciprocating the plug body in the conduit at said region.

11. A method according to claim 9 or 10 in which the plug body comprises an active treatment agent.

12. A method according to claim 11 in which the treatment agent is a cleaning agent such as a biocide or bleach.

13. A method according to claim 9, 10, 11 or 12 comprising detecting the arrival or presence of the plug body at said region.

14. A method of cleaning, clearing or separating in a fluid flow conduit by causing a body of the agglomerate material to pass along inside the conduit, spanning its interior to contact its interior surface, characterised in that the body of the material is passed along the conduit adjacent a gas-filled void, the void being between said body and another such body, or between said body and other flowable material in the conduit.

15. A method according to claim 14 in which the leading face of the moving body is a said face at the interface with the adjacent void.

16. A method according to claim 14 or 15 in which agglomerate material is passed along the conduit as two or more portions separated by one or more corresponding voids.

17. A method of cleaning, treating, clearing or providing a movable barrier in a conduit, comprising passing along inside the conduit a plug body of a coherent flowable agglomerate material consisting essentially of cohering particles of frozen solid material mixed with a liquid which comprises a melt liquid corresponding to the frozen material of the solid particles,

characterised in that

the particles of frozen solid are structured with a gradation of melting point from the inside to the outside of the particles.

18. A method according to claim 17 in which the particle material decreases in freezing point from the inside to the outside and the surrounding liquid has a still lower freezing point.

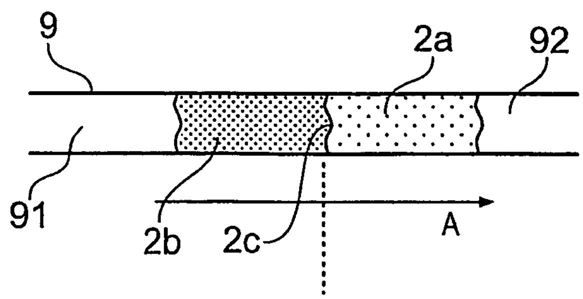


Fig. 1

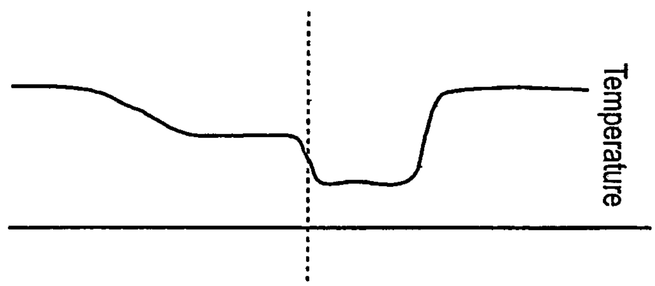


Fig. 2

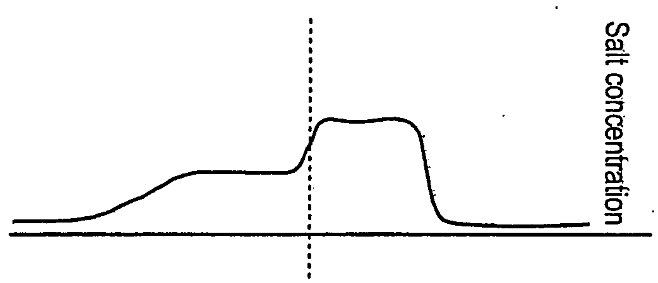


Fig. 3

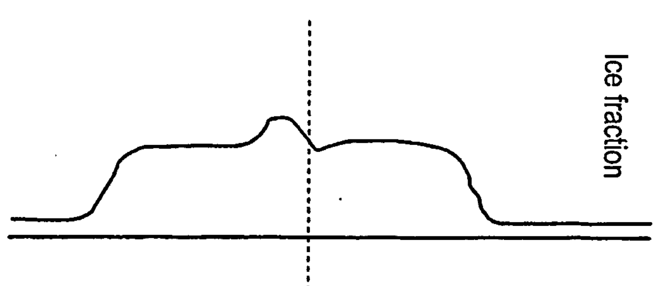


Fig. 4

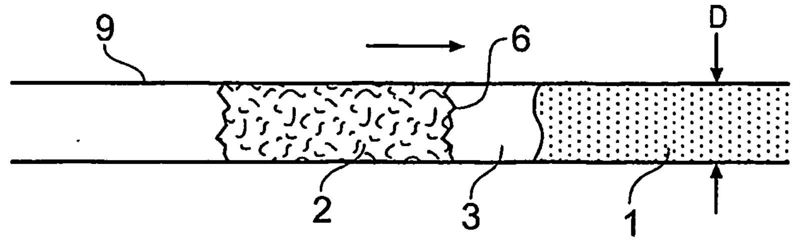


Fig. 5

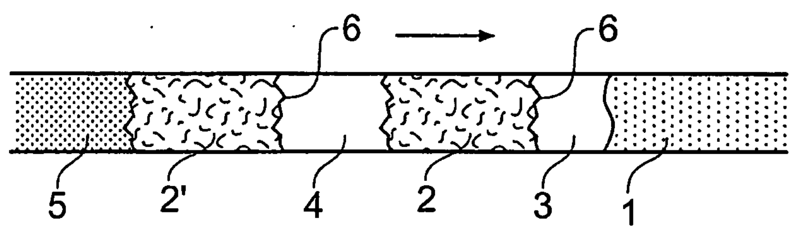


Fig. 6

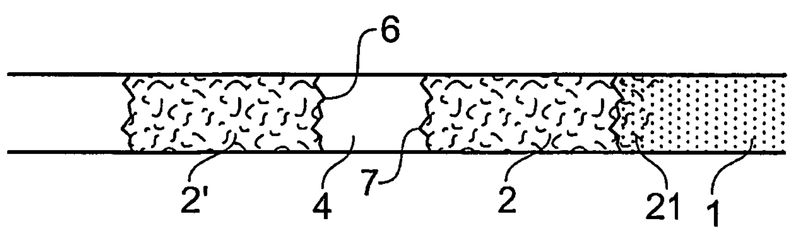


Fig. 7

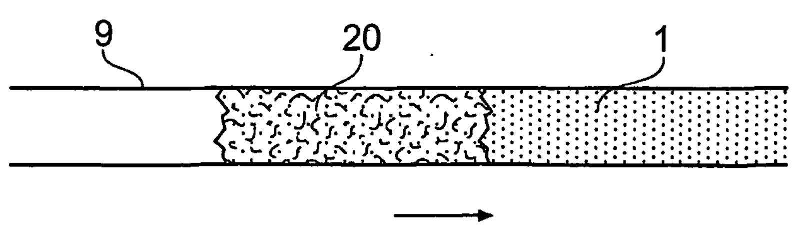


Fig. 8

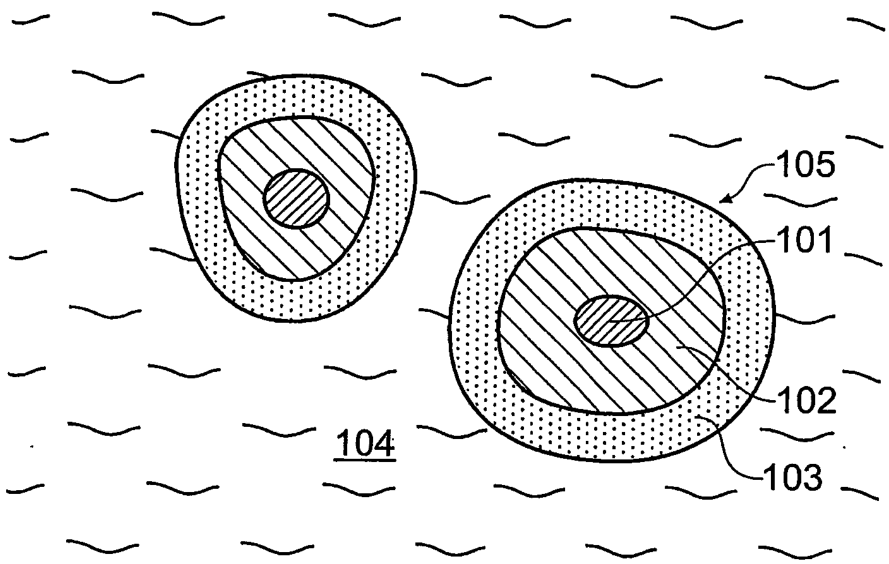


Fig. 9

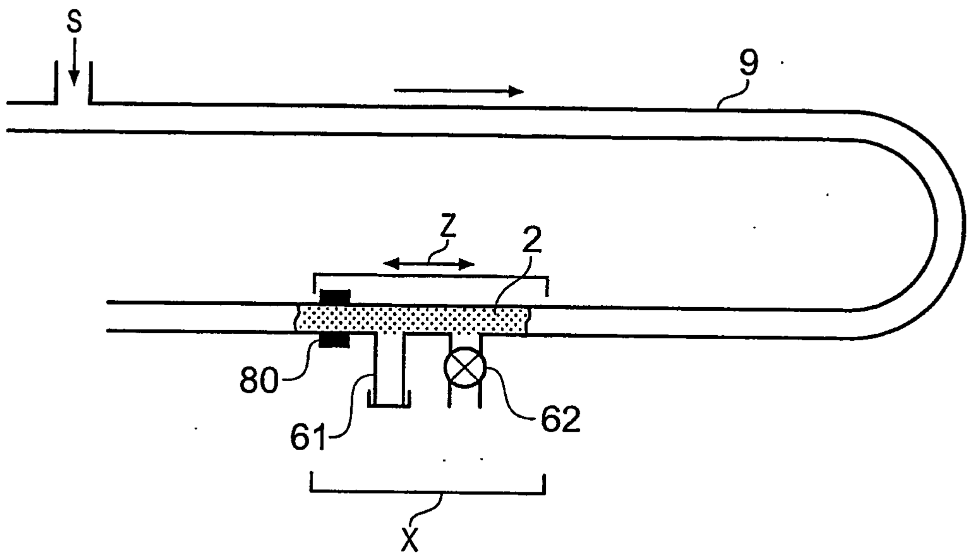


Fig. 10

# INTERNATIONAL SEARCH REPORT

Internat pplication No
PCT/GB 03/02697

**A. CLASSIFICATION OF SUBJECT MATTER**
IPC 7 B08B9/055 F17D3/08

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B08B F17D F28G B67D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category ° | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| Y | WO 01 51224 A (UNIVERSITY OF BRISTOL ET AL) 19 July 2001 (2001-07-19) cited in the application | 9-13 |
| A | the whole document --- | 1,3-8, 14,17 |

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

| Date of the actual completion of the international search | Date of mailing of the international search report |
|---|---|
| 13 October 2003 | 20/10/2003 |

| Name and mailing address of the ISA | Authorized officer |
|---|---|
| European Patent Office, P.B. 5818 Patentlaan 2<br>NL – 2280 HV Rijswijk<br>Tel. (+31–70) 340–2040, Tx. 31 651 epo nl,<br>Fax: (+31–70) 340–3016 | van der Zee, W |

# INTERNATIONAL SEARCH REPORT

Internati... ...plication No
PCT/GB 03/02697

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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## INTERNATIONAL SEARCH REPORT

Inten al application No.
PCT/GB 03/02697

**Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)**

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. [ ] Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. [ ] Claims Nos.:
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:

3. [ ] Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

**Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)**

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. [ ] As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. [X] As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. [ ] As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. [ ] No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

**Remark on Protest**

[ ] The additional search fees were accompanied by the applicant's protest.

[ ] No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

| Patent document cited in search report | | Publication date | Patent family member(s) | | | Publication date |
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